STUDY OF PHYSICAL-CHEMICAL AND COLLOIDAL PROPERTIES OF THE QUATERNARY AMMONIUM SALT OBTAINED FROM HEPTADECANOIC AND OCTADECANOIC ACIDS WITH TRIETHANOLAMINE

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Abstract: *Currently, surface-active agents (surfactants) have found widespread application in various industries, including detergents, foaming agents, emulsion stabilizers, fluorogens, hydrophobizers, and corrosion inhibitors. As a result, the synthesis of surfactants has become a critical area of study. This paper presents the results of an investigation into the oil-repelling and oil-dispersing properties of complex salts formed by heptadecane (HA) and octadecane (OA) acids with triethanolamine (TEA), in distilled, potable, and seawater contaminated with Balakhani oil. The study measured the surface activity of these complex salts at various concentrations using a tensiometer, and the elemental composition was determined through calculation methods. Infrared spectroscopy was employed to analyze the composition of the newly synthesized complex salts, confirming that the reaction followed the proposed scheme. The comparative study of the complex salts formed by HA and OA with TEA revealed that the OA - TEA complex salt demonstrated superior oil-dispersing properties, particularly in potable and seawater. Specifically, the HA-TEA complex salt reduced surface tension from 72.0 mN/m to 33.3 mN/m, whereas the OA-TEA complex salt reduced surface tension from 72.0 mN/m to 29.4 mN/m, indicating its higher surface activity. The elemental composition of the complex salts formed by both acids with TEA was calculated and revealed the following mass fractions: for HA-TEA (%): C 65.8, H 11.7, O 19.1, N 3.4; for OA-TEA (%): C 66.4, H 11.8, O 18.5, N 3.3. Solutions of the complex salts in various concentrations exhibited a mixed effect when tested in seawater contaminated with Balakhani oil. Both complex salts were soluble in ethyl and isopropyl alcohols. The study also highlighted that the OA-TEA complex salt, in particular, displayed promising characteristics in terms of its composition and performance in environmental applications.*

Key words: *oil recovery, oil dispersing, surface tension, surfactant, ammonium salts*

Introduction

A surfactant is a chemical compound that reduces surface tension by concentrating at the interface between thermodynamic phases [1]. Surfactants are among the most widely used and commercially significant chemical reagents in both industry and everyday life. Surfactants are widely used as emulsifiers, wetting agents, foaming agents, dispersants, and antistatic additives [2-4]. Natural and synthetic surfactants play a key role in various processes, primarily by reducing the surface tension between liquid and solid phases [5-8]. Their importance has led to widespread use in various industries [9] and in everyday life [6-10]. For example, in the food [11], cosmetics [12], and pharmaceutical industries [13], surfactants help stabilize emulsions; in household products [11] such as soaps [1], shampoos [2-3], hand sanitizers [14-15], and cleaning agents, they help remove contaminants from water [16-17]. Surfactants used to remove thin oil films from water are divided into oil-dispersing and oil-repellent [5-7, 12-14, 16, 17]. This article is devoted to the study of the physicochemical properties, as well as oil-dispersing and oil-repellent properties, of a complex salt formed by heptadecanoic and octadecanoic acids with triethanolamine (TEA).

Experimental part

Heptadecane acid ($C_{16}H_{33}COOH$, HA) is a saturated fatty acid with a molecular mass of 270.5 g/mol. It is solid at room temperature, odorless, white, and insoluble in water but soluble in organic solvents like ether and benzene. The compound has a melting point of 61°C and a boiling point of 227°C. Octadecane acid ($C_{17}H_{35}COOH$, OA), another saturated monobasic fatty acid, has a molecular mass of 284.5 g/mol. It is also solid at room temperature, odorless, white, and insoluble in water but soluble in solvents such as ether, chloroform, and benzene. Its melting point is 70°C, and its boiling point is 361°C.

TEA, with a molecular mass of 149.19 g/mol, is a colorless, transparent liquid with an ammonia-like odor. It has a density of 1.124 g/ml, a freezing point of 22°C, a boiling point of 335°C, and a refractive index of 1.4850 at 20°C. TEA is produced by the "Kazanorgsintez" Joint-Stock Company in Russia, and OA ($C_{17}H_{35}COOH \geq 97\%$) is produced by Sigma Aldrich.

The surface activity of the substances was measured at the air-water interface using the Du Nouy ring method with a KSV Sigma 702 (Finland) tensiometer. The experiments involved light Ramana oil (density ρ20 = 0.862 g/cm³, kinematic viscosity ν_{20} = 16.80 cSt) as well as distilled, potable, and seawater.

The properties of potable water at 20°C: density: 0.996 g/cm³, pH: 7-8. Chemical composition (in gram equivalents per 100 g of water): Ca^{2+}- 0.0052; Mg^{2+}- 0.0023; Cl^{-} -0.0007; SO_4^{2-} - 0.0044; HCO_3^{-} - 0.0273; CO_3^{2-} - 0.0009; total hardness: 4.5 mg-equivalent/g.

The properties of seawater at 20°C: density: 1.0098 g/cm³, pH: 7.7. Chemical composition (in mg per liter of water): Na^{+} - 2650; K^{+} -20; Ca^{2+} - 250; Mg^{2+} - 900; NH_4^{+} - 0.15; Cl^{-} - 500; SO_4^{2-} - 2800; NO_3^{-} - 0.1; PO_4^{3-} - 0.35; SiO_2 - 0.5; total hardness: 69 mg-equivalent/g.

Conducting the research.

The reaction between OA and TEA under the conditions you mentioned (1:1 molar ratio, 70-80°C, and intensive mixing for one day) would indeed lead to the formation of a complex salt or surfactant. Here's the detailed reaction scheme and mechanism based on the conditions described:

$$C_{17}H_{35}COOH + N(C_2H_4OH)_3 \rightarrow [C_{17}H_{35}COO^{-} N^{+} H(C_2H_4OH)_3]$$

The reaction between OA and TEA yields a quaternary ammonium salt that appears as a soft, wax-like solid with a pale yellow color at room temperature. This complex salt is characterized by the following properties:

- **Molecular mass:** 433.67 g/mol.
- **Solubility:** The salt is readily soluble in organic solvents such as ethanol and isopropanol, indicating its amphiphilic or predominantly non-polar character.
- **Elemental composition (found, %):** C – 66.4; H – 11.8; O – 18.5; N – 3.3.

These elemental ratios confirm that the compound's structure is largely defined by the long hydrocarbon chain derived from octadecane acid, the nitrogen atom provided by triethanolamine, and oxygen atoms originating from the carboxyl group and potential ester linkages. Such a molecular arrangement is typical of surfactants, which feature hydrophobic alkyl tails (carbon chains) combined with hydrophilic head groups containing nitrogen and oxygen functionalities.

The reaction between HA and TEA at a 1:1 molar ratio, under the conditions of 65-70°C with intensive mixing for one day, would indeed lead to the formation of a complex salt. The carboxyl group of HA reacts with the amine group of TEA, resulting in the formation of a quaternary ammonium salt.

The reaction can be simplified as below:

$$C_{16}H_{33}COOH + N(C_2H_4OH)_3 \rightarrow [C_{16}H_{33}COO^{-} N^{+} H(C_2H_4OH)_3]$$

The product of this reaction is a quaternary ammonium salt with a structure similar to the one formed between OA and TEA but with the longer carbon chain of HA.

The product is likely a soft, wax-like solid at room temperature. It dissolves in solvents like alcohols (e.g., ethyl and isopropyl alcohol). The salt will have a long hydrophobic chain (from the HA) and a hydrophilic head (from the TEA).

The quaternary ammonium salt formed from HA and TEA has the following characteristics: Molecular Mass: 419.6 g/mol. Solubility: This salt dissolves well in solvents like ethyl alcohol and isopropyl alcohol. Found, %: C 65.8; H 11.7; O 19.1; N 3.4.

A comparative analysis of the elemental compositions of the complex salts formed from HA and OA with TEA reveals a difference in the mass fraction of carbon. The quaternary ammonium salt formed from OA and TEA exhibits a higher mass fraction of carbon compared to the salt formed from HA. This is because OA contains a longer hydrocarbon chain (C_{17}), which leads to a slightly higher carbon content in the resulting complex salt. HA has a C_{16} carbon chain, whereas OA has a C_{17} carbon chain, causing a subtle difference in the mass fraction of carbon in the final complex salts. Despite the difference in carbon content, both salts share common properties, such as their solubility in alcohols and their wax-like solid state at room temperature.

Results and discussion

The structure of the complex salts formed by HA and OA with TEA was analyzed using IR spectroscopy. IR spectra can provide important information about the functional groups and bonding in the compound (Fig.s 1 and 2).

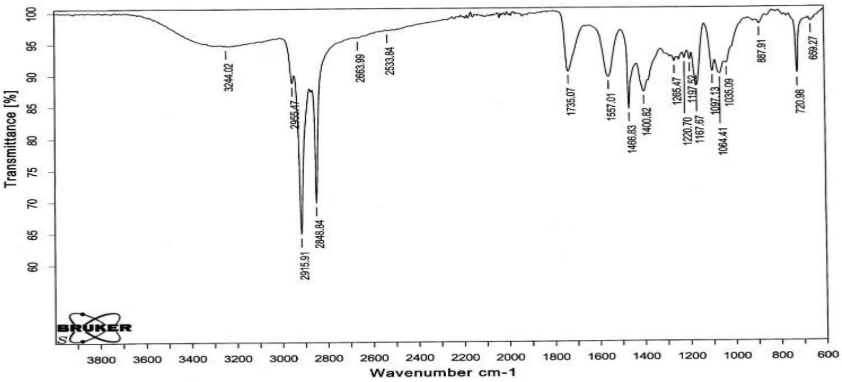

Fig. 1. The IR spectrum of the complex salt formed by OA and TEA

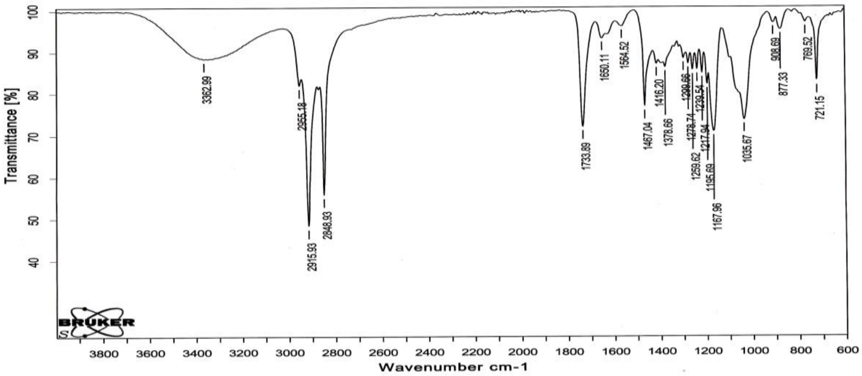

Fig. 2. The IR spectrum of the complex salt formed by HA and TEA

When HA and OA react with TEA to form quaternary ammonium salts, the IR spectrum shows the formation of a carboxyl ion ($-COO^{-}$), with a carbonyl signal in the 1650-1550 cm^{-1} range. The characteristic C-N stretching signals of the quaternary ammonium salts are observed in the 1250-100 cm^{-1} range, indicating that the reaction follows the above scheme (Fig.s 1 and 2).

The values of the specific electrical conductivity of aqueous solutions of various concentrations of the complex salts formed by HA (1) and OA (2) with TEA were determined using a conductivity meter (Fig. 3). As seen in the figure, as the concentration of SAMs in the solution increases, the values of the specific electrical conductivity also increase.

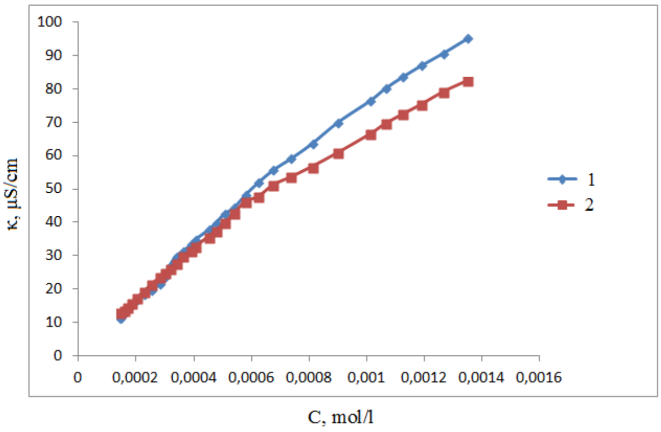

Fig. 3. Results of the study of the specific electrical conductivity of aqueous solutions with various concentrations of the complex salts formed by HA (1) and OA (2) acids with TEA

The surface activity of various concentrations of the complex salts formed by HA and OA with TEA was determined at a temperature of 21°C at the air-water interface using a tensiometer (Table 1).

Table 1. Results of the study of surface activities of complex salts formed by HA and OA with TEA at the air-water interface (t = 21°C).

Name of the sample	**SAM concentration (% by weight)**											
	0.00025	0.0005	0.00075	0.001	0.0025	0.005	0.0075	0.01	0.025	0.05	0.075	0.1
	Surface tension values at the air-water interface, $mN \cdot m^{-1}$											
HA + TEA	59.8	52.0	48.5	46.4	41.4	38.2	36.5	35.4	33.9	33.6	33.4	33.3
OA + TEA	65.6	55.7	50.0	46.1	36.9	33.5	32.2	31.0	30.1	29.4	29.7	29.4

Based on the surface tension values measured using the tensiometer, surface tension isotherms were plotted in the $\gamma - \ln C$ coordinates (Fig. 4).

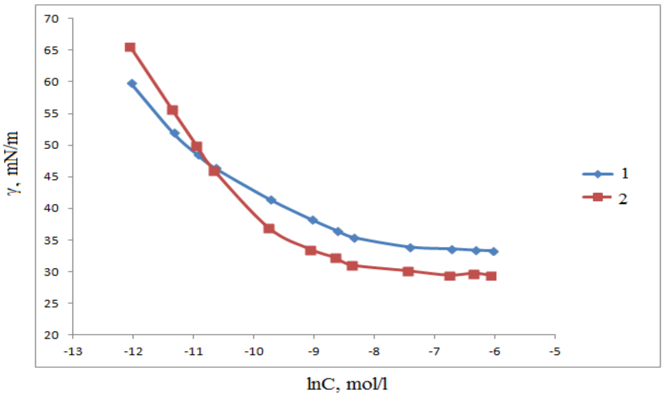

Fig. 4. Surface tension isotherms of complex salts formed by HA (1) and OA (2) fatty acids with TEA

Based on this figure, the value of $d\gamma/d\ln C$ has been determined using graphical methods. In a reagent-free environment, the surface tension at the water-air interface is 72.0 $mN \cdot m^{-1}$. The stabilization of surface tension values for the SAM formed from the complex salts of HA and OA fatty acids with TEA occurs at 33.9 and 29.4 $mN \cdot m^{-1}$, respectively. The values of maximal adsorption (G_{max}) were calculated based on the interphase surface tension values using the Gibbs equation [18]:

$$G_{max} = -\frac{1}{nRT} \lim_{C \to C_{CMC}} \left(\frac{\partial \gamma}{\partial \ln C}\right)_T \qquad (1)$$

In this context, $d\gamma/d\ln C$ represents the slope of the isotherm curve at concentrations lower than the critical micelle concentration (CMC) at a constant temperature; R is the universal gas constant (8.314 $J \cdot mol^{-1} \cdot K^{-1}$); n is the number of ions formed during the dissociation of the SAM.

The area per SAM molecule at the interphase surface – the minimal surface area (A_{min}) - provides information about the degree of orientation of these molecules. The average minimal surface area is calculated using the following equation [19]:

$$A_{min} = \frac{1}{G_{max} N_A} \qquad (2)$$

In this context, N_A is Avogadro's number (6.023×10^{23}).

The efficiency value (pC_{20}) is determined at the concentration (mol/l) where the interfacial tension of the SAM decreases to 20 mN/m [20]:

$$pC_{20} = -\lg C_{(-\Delta\gamma=20)} \qquad (3)$$

The surface pressure or efficiency (π_{CMC}) at the water-air interface of aqueous solutions of the synthesized substances is determined:

$$\pi_{KMQ} = \gamma_0 - \gamma_{KMQ} \qquad (4)$$

Here, γ – is the surface tension in a medium without SAM, and γ_{CMC} is the surface tension of the solution at the critical CMC.

For ionogenic SAMs, the thermodynamic parameters of the micelle formation (ΔG_{mis}) and adsorption (ΔG_{ad}) processes have been calculated using the Gibbs equations:

$$\Delta G_{mis} = (2-\alpha)\, RT \ln KMQ \qquad (5)$$

Here, X_{KMQ} is the concentration of SAM at the CMC point. $X_{KMQ} = KMQ/55.4$.

$$\Delta G_{ad} = (2-\alpha)\, RT \ln KMQ - 0.6023 \pi_{KMQ} A_{KMQ} \qquad (6)$$

Table 2 shows the colloidal chemical parameters of the synthesized reagents, calculated using formulas (1-6).

Table 2. Colloid-chemical parameters of the complex salts formed by HA and OA fatty acids with TEA

SAM	**β**	**$KMQ \times 10^{-3}$, $mol \cdot dm^{-3}$**	**$G_{max} \times 10^{-10}$, $mol \cdot sm^{-2}$**	**$A_{min} \times 10^{-2}$, nm^2**	**γ_{KMQ}, $mN \cdot m^{-1}$**	**π_{KMQ}, $mN \cdot m^{-1}$**	**pC_{20}**	**ΔG_{mis}, kC/mol^{-1}**	**ΔG_{ad}, kC/mol^{-1}**
HA	0.36	0.59	2.13	75.1	33.9	38.1	4.93	-37.80	-55.02
OA	0.31	1.15	2.19	75.8	29.4	42.6	4.82	-34.33	-53.78

Note: β - the degree of association of the counter-ion, CMC - critical micelle concentration, γ_{CMC} - surface tension of the solution at CMC, G_{max} - maximal adsorption, A_{min} - the minimal surface area of the polar group, π_{CMC} - surface pressure or efficiency, pC_{20} - efficiency value, ΔGmis - enthalpy change during micelle formation, ΔGad - enthalpy change during the adsorption process.

The study was conducted under controlled laboratory conditions to evaluate the oil-collecting and oil-dispersing performance of the quaternary ammonium salts formed by heptadecane acid (HA) and octadecane acid (OA) with triethanolamine (TEA). These surfactant-type compounds were tested for their potential application in purifying water surfaces contaminated by thin layers of Balakhani light oil.

Methodology. *Water samples:* Three types of water were used—distilled water, drinking water, and seawater of varying mineralization.

Oil layer formation: A 1 mL sample of Balakhani light oil was carefully introduced to each water surface, forming a uniform layer with a thickness of approximately 0.17 mm.

Reagents: Both the pure quaternary ammonium salts and their 5 % aqueous solutions were evaluated. The amount of reagent applied was systematically varied to assess dosage effects.

Application process: After the oil layer was allowed to spread evenly, the predetermined amount of reagent was added to the water surface.

Observation and measurement: The effectiveness of each reagent was determined by monitoring the decrease in oil-film area over time, continuing until the oil was either completely dispersed or collected.

The oil-collecting coefficient (K) was calculated as the ratio of the initial area of the oil layer to the area of the oil stain formed after the reagent's effect. This coefficient provides a quantitative measure of the reagent's effectiveness in collecting or dispersing the oil. The oil-collecting coefficient (K) is an important indicator of how effective the complex salt is in either collecting or dispersing the oil. A higher value of K means the reagent was more effective in reducing the area of the oil stain (i.e., better at collecting the oil). The study observed the effectiveness of both pure complex salts and 5% aqueous solutions of the salts in different water types (distilled, drinking, and seawater). The data on the oil-collecting efficiency were presented in Table 3, showing how the reagent performed across different water samples and varying concentrations of the complex salts. The reduction in the oil stain area indicated the oil-collecting capacity of the complex salt, helping to assess its potential for practical use in cleaning oil spills. The complex salts formed by heptadecane and octadecane acids with TEA show promising oil-collecting properties, as indicated by the reduction in the oil stain area. Both pure complex salts and aqueous solutions were effective in the oil dispersion and collection processes. The degree of effectiveness likely varied with the water type (distilled, drinking, or seawater) and concentration of the reagent. This study suggests that the complex salts could be used in real-world scenarios such as cleaning water surfaces contaminated with oil, as they show a significant ability to collect or disperse the oil efficiently.

Table 3. Research results of the oil-collecting and oil-dispersing capabilities of the HA (OA) and TEA complex salt (Ramana oil; thickness 0.17 mm)

Reagent applied to the surface of the oil	**Distilled water**		**Drinking water**		**Sea water**	
	τ, hours	**Disp.K(K_D,%)**	**τ, hours**	**Disp.K(K_D,%)**	**τ, hours**	**Disp.K(K_D,%)**
HA and TEA						
Reagent's 5% aqueous dispersion	0	8.2	0	7.8	0-4.0	11.1
	1.0-42.0	15.2	6.0-20.0	72.2	20.0-44.0	82.2
	70.0	10.2	44.0	76.8	60-84.0	76.8
OA and TEA						
In pure form	1.0-5.0	70.1	1.0-5.0	63.7	1.0-5.0	72.6
	21.0-76.0	86.8	21.0-76.0	75.0	21.0-76.0	65.9
Reagent's 5% aqueous dispersion	1.0-5.0	75.0	1.0-5.0	72.0	1.0-5.0	78.6
	21.0-76.0	79.2	21.0-76.0	82.4	21.0-76.0	86.8

As seen from Table 3, the oil-collecting capability of the complex salt formed by HA and TEA in a 5% solution of the reagent is $K_{max} = 15.2$ mN/m. The reagent exhibits a mixed effect as an oil-collecting and dispersing agent in drinking and seawater (with $K_{max} = 7.8$ and 11.1; $K_D = 76.8\%$

and 82.2%, respectively). The reagent maintains its effect for a duration of 4 days.

As seen from Table 3, the complex salt formed by OA with TEA, both in its pure form and in 5% solutions, exhibits oil-dispersing properties in both drinking and seawater.

The study revealed that, like other higher carboxylic acids [21], the complex salts formed by OA and HA with TEA are also surfactants.

Conclusion

1. Specifically, the HA-TEA complex salt reduced surface tension from 72.0 mN/m to 33.3 mN/m, whereas the OA-TEA complex salt reduced surface tension from 72.0 mN/m to 29.4 mN/m, indicating its higher surface activity.
2. The complex salts formed by OA and HA with TEA are well soluble in polar solvents such as water, ethyl, and isopropyl alcohol. This is due to the fact that they contain both hydrophobic and hydrophilic groups.
3. The complex salts formed by these acids with TEA can be used as oil-collecting and oil-dispersing reagents. The complex salts formed by these acids with TEA can be used as oil-collecting and oil-dispersing reagents.
4. The study found that the TEA complex salt of OA exhibits promising properties, especially in terms of composition and ecology.

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